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THERMAL ANALYSIS OF A LOW-GRADE PHOSPHATE SAMPLE ON BEHALF OF ESSO RESOURCES CANADA LIMITED

W.S. Bowman
Extractive Metallurgy Laboratory

MICROMEDIA

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(This report relates essentially to the sample as received)

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CONFIDENTIAL

THERMAL ANALYSIS OF A LOW-GRADE PHOSPHATE SAMPLE ON BEHALF OF ESSO RESOURCES CANADA LIMITED

by

W.S. Bowman*

A sample of a Canadian low-grade phosphate deposit was received from Esso Resources Canada Limited for thermogravimetry (TG) and differential thermal analysis (DTA) in inert and oxidizing atmosphere in order to detect the presence of carbonate and oxidizable minerals.

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*Technologist, Physical Chemistry Section, Extractive Metallurgy Laboratory, Mineral Sciences Laboratories, CANMET, Energy, Mines and Resources Canada.

EXPERIMENTAL

The samples for TG, weighing about 300 mg, were placed unpacked in a 2.6 mL platinum crucible. The thermobalance was a Stanton MF-S1 in which the crucible rests on a pedestal above the balance and is surrounded by a mullite protection tube and a platinum/rhodium-wound furnace. For air atmosphere, the gas inlets and outlets were left open to the atmosphere. For inert atmosphere, argon was flushed through the balance and protection tube at 150 mL/min. Nominal heating rate was 6°C/min. Sample weight and temperature were recorded simultaneously on a two-pen recorder.

The samples for DTA, weighing about 0.5 g, were lightly packed into a cavity in a palladium sample block. The cylindrical cavity was 6 mm in diameter and 12 mm deep. An identical cavity, 3 mm from the former, contained the reference material, α-alumina. Pt vs Pt: 13% Rh thermocouples were imbedded in the sample and reference through holes drilled centrally into the wall of each cavity from one end of the holder. The entire arrangement was inserted into a mullite protection tube in a Kanthal-wound furnace. The gas inlet and outlet were either left open to the atmosphere or suitable gas was flushed through at 150 mL/min. A nominal heating rate of 12°C/min was used. The temperature (T) was measured from the reference thermocouple and the difference in temperature between the sample and reference thermocouples (ΔT) was amplified with a Leeds and Northrup 9835-B Microvolt Amplifier. These two signals were recorded on a Leeds and Northrup Speedomax W/L two-pen recorder equipped with a feature that permitted automatic biasing of the latter signal to keep it on scale.

RESULTS

Copies of the TG and DTA charts reduced to 8½ by 11 inches are appended to this report. On the TG charts, the temperature vs time curve is on the right with the scale increasing from right to left. The sample weight vs time curve is on the left with the scale increasing from left to right (0 to 100 mg). The horizontal lines are caused by automatic momentary arrestment of the balance every 5 min.

On the DTA charts, the temperature scale is indicated. The ΔT scale is -100μV to +100μV with zero in the centre of the scale. In one case the scale was biased by approximately 100μV to keep the pen on scale.

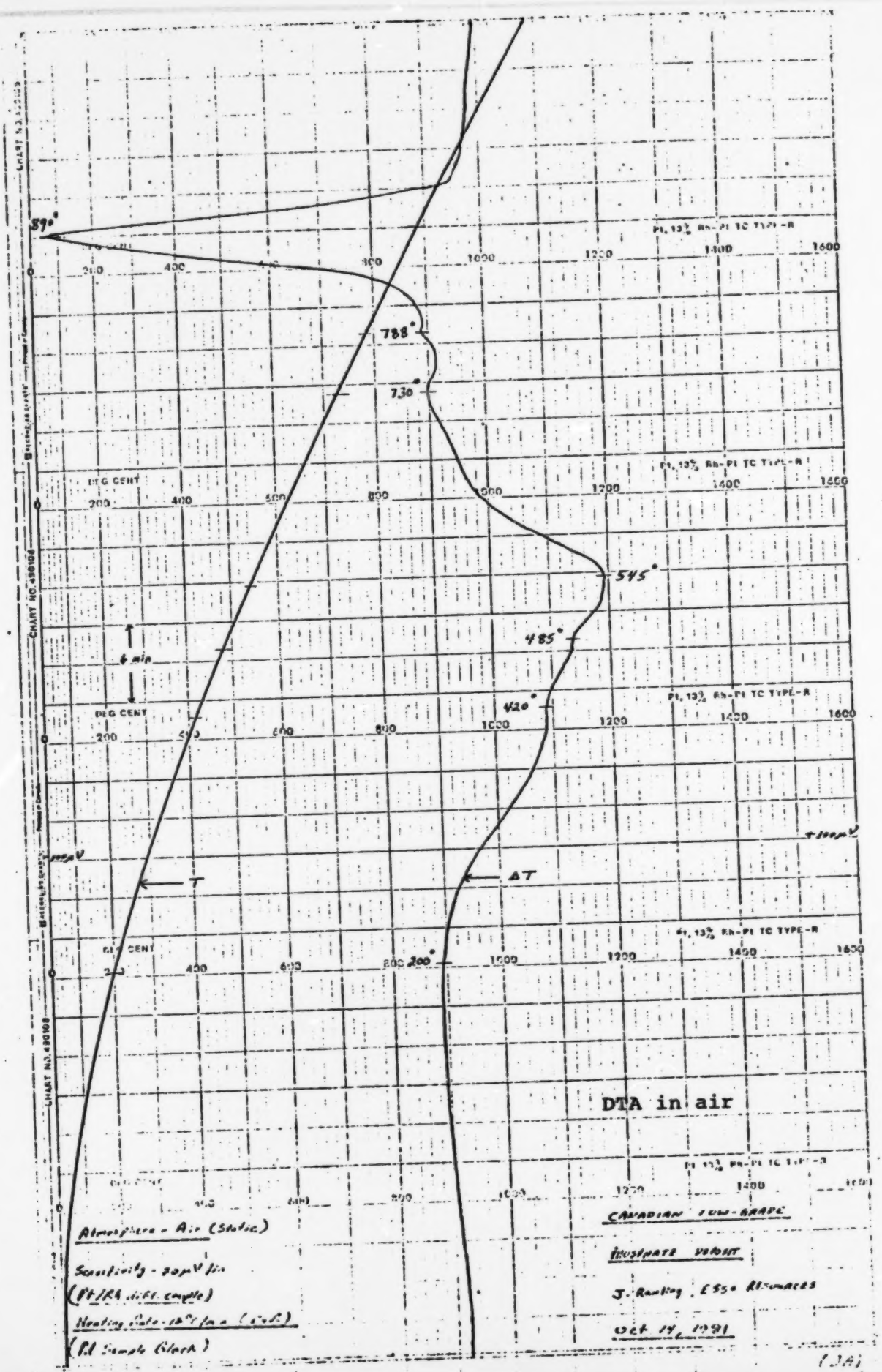
There is a large endothermic DTA peak at 870°C in Ar, 890°C in air and 932°C in CO_2. This peak is undoubtedly due to the decomposition of calcite. The CO_2 loss is estimated from the TG charts to be 13.4% in Ar and 12.1% in air. This corresponds to roughly 29% $CaCO_3$.

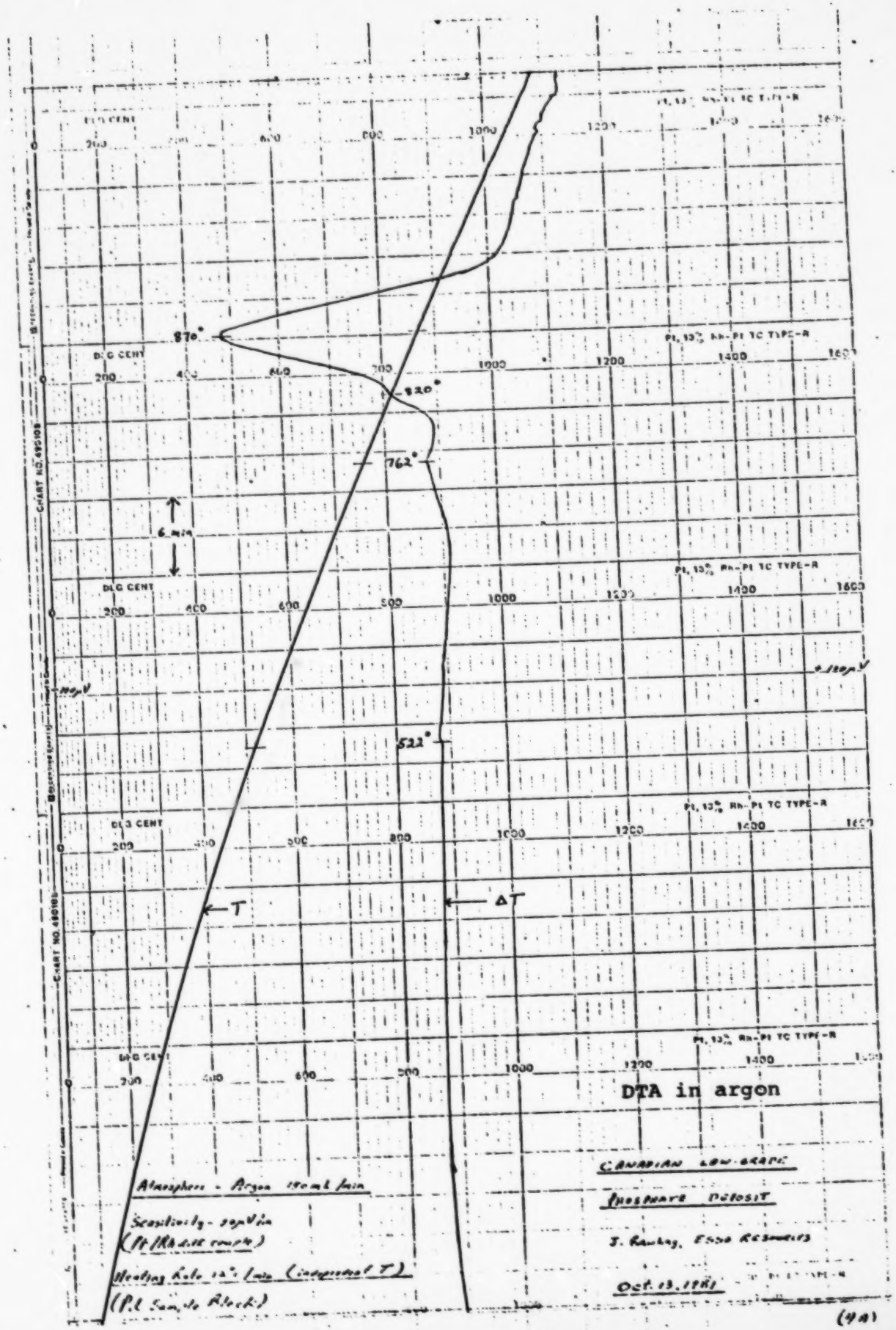
The DTA chart for the sample run in air shows a series of broad exothermic peaks from about 200°C to 700°C which were absent in the argon and CO_2 runs. There was a corresponding weight loss of about 2% on the TG run in air. This effect could be due to oxidation of material such as iron sulphide (FeS_2) or a small amount of organic material or both.

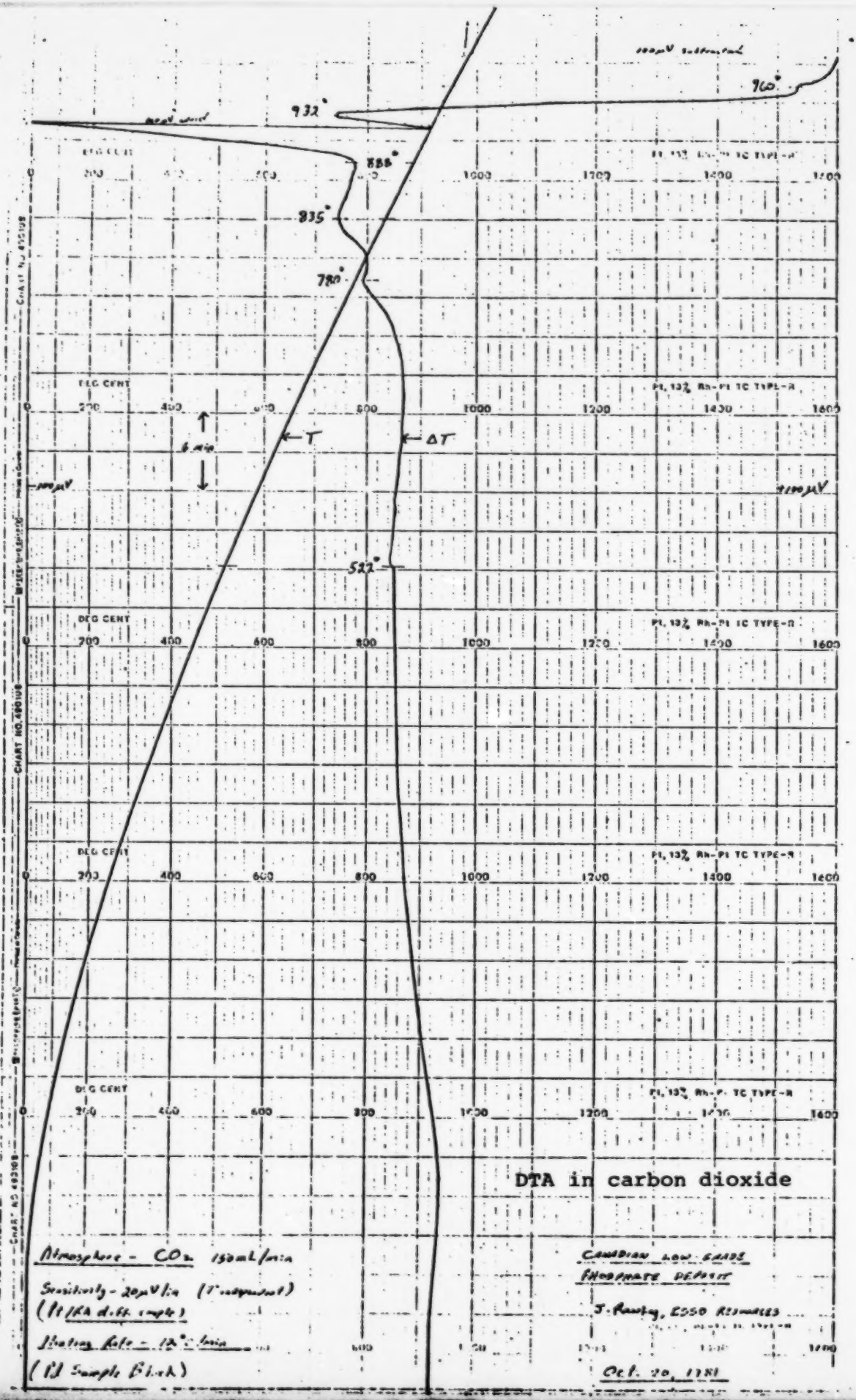
There is a small endothermic DTA peak at 788°C in air (762°C in Ar and 780°C in CO_2) that could be due to loss of combined water.

There is also an endothermic peak at 820°C in Ar and 835°C in CO_2 which is absent in the chart made in air. This peak could result from the decomposition of some material that underwent oxidation in air, such as the pyrolysis of organic material. The weight change that would correspond to these two peaks could not be resolved from the large weight loss due to calcite decomposition.

There is no weight loss or DTA peak before 120°C, which indicates that the sample is moisture-free. No explanation is offered for the bump on the DTA curves at 522°C in Ar and CO_2.


570°
788°
730°
545°
485°
420°
200°
6 min
DEG CENT
T
ΔT
DTA in air
Atmosphere - Air (Static)
Sensitivity - 20μV/in
(Pt/Rh diff. couple)
Heating Rate - 10°C/min (10°C)
(Pt Sample Block)
CANADIAN LOW-GRADE
PHOSPHATE VOLCLIT
J. Rowley, Esso Resources
Oct 14, 1981
(JA)


870°
820°
762°
522°
6 min
T
ΔT
DEG CENT
Pt, 13% Rh-Pt TC TYPE-R
DTA in argon
CANADIAN LOW-GRADE
PHOSPHATE DEPOSIT
J. Rawlay, ESSO RESOURCES
Oct. 13, 1961
Atmosphere - Argon 100 ml/min
Heating Rate 10°/min (Isothermal T)
(Pt Sample Block)



DTA in carbon dioxide

Atmosphere - CO_2 150 ml/min

Sensitivity - 20 μV/in (T measurement)
(Pt/Rh diff. couple)

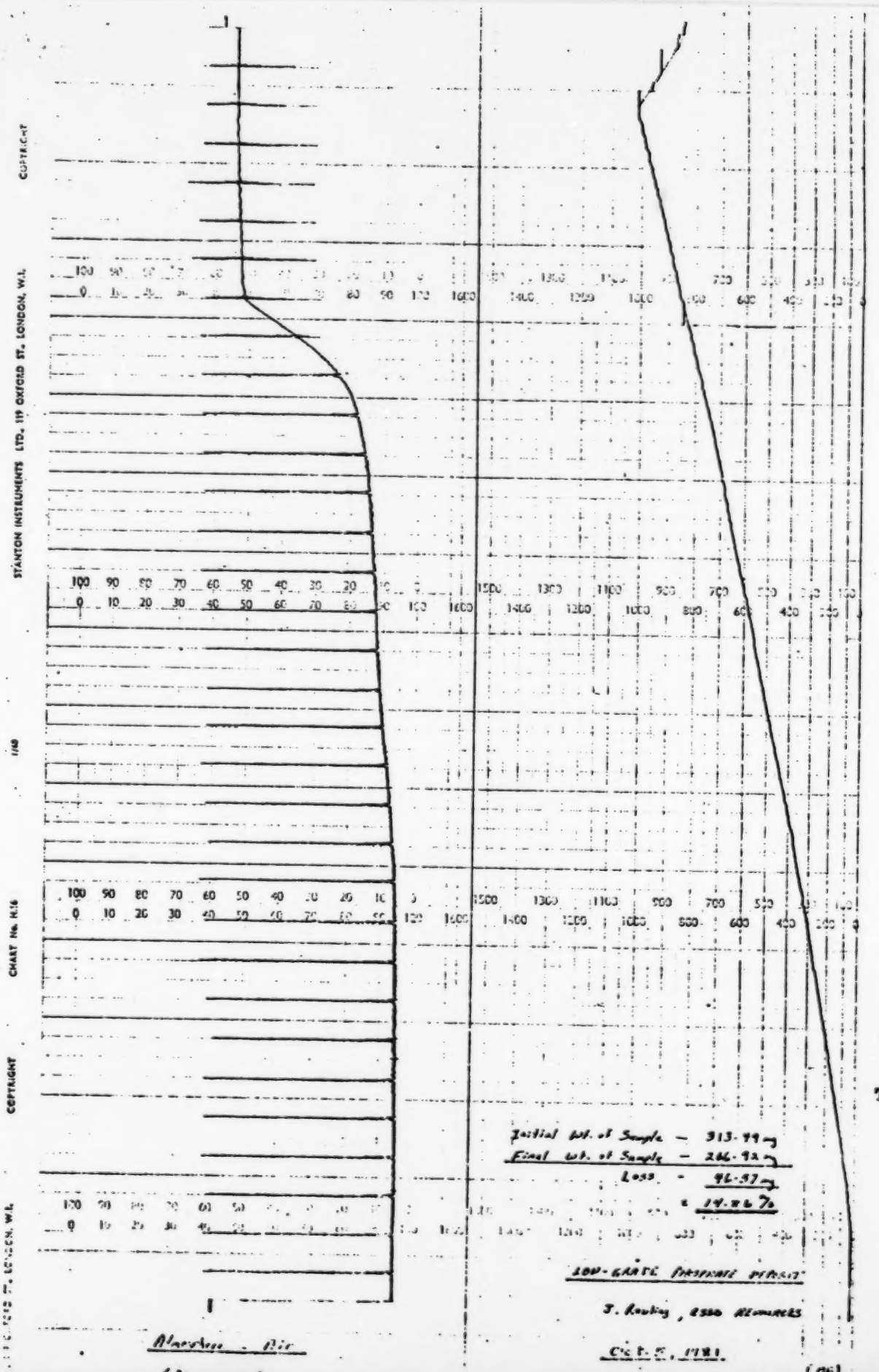
Heating Rate - 12°C/min

(Pd Sample Block)

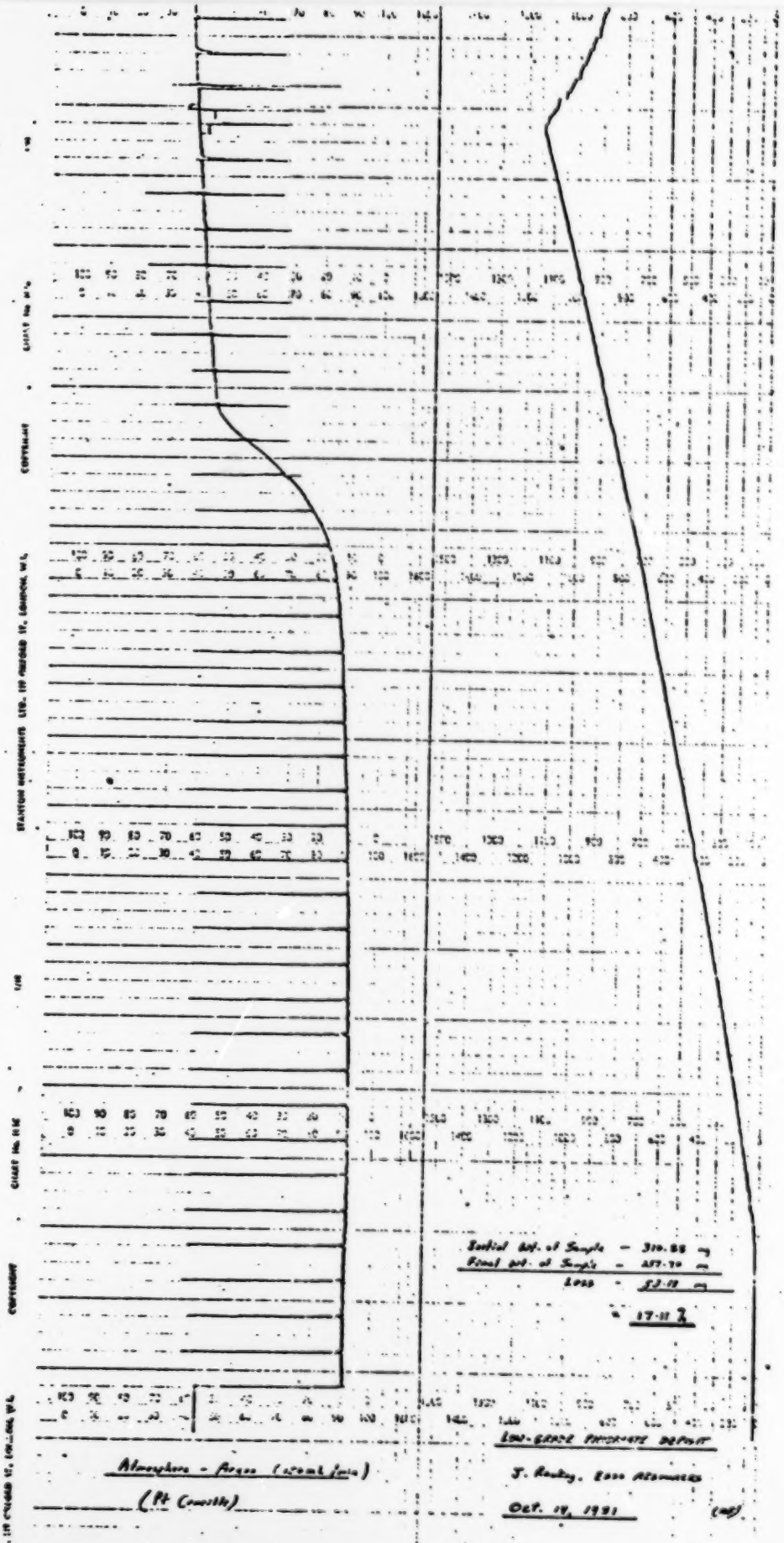
CANADIAN LOW GRADE
PHOSPHATE DEPOSIT

J. Rowley, ESSO RESOURCES

Oct. 20, 1971


COPYRIGHT
STANTON INSTRUMENTS LTD., 119 OXFORD ST., LONDON, W.1.
CHART No. H.16
COPYRIGHT
Initial wt. of Sample – 313.99 mg
Final wt. of Sample – 266.92 mg
Loss – 46.57 mg
= 14.76 %
100-GRADE PHOSPHATE PERMIT
J. Rowley, ESSO RESEARCH
Oct. 5, 1961.
Atmosphere - Air
(N Sample)

TG in air


Initial Wt. of Sample – 310.88 mg
Final Wt. of Sample – 257.70 mg
Loss – 53.18 mg
17.11 %
Low-Grade Phosphate Deposit
J. Rooney, Esso Minerals
Oct. 19, 1981
Atmosphere – Argon (zero leak)
(Pt Crucible)

TG in argon